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Indian Standard

CODE OF PRACTICE FOR HOT-DIP ALUMINIZING OF IRON AND STEEL

(First Revision)

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0. FOREWORD

- 0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 24 October 1988, after the draft finalized by the Hot Dip, Sprayed and Diffusion Coatings Sectional Committee had been approved by the Structural and Metals Division Council.
- **0.2** This standard was first published in 1977. The present revision has been prepared in the light of the experience gained since its first publication. In this revision, clauses pertaining to rinsing, adhesion test and safety measures have been modified.
- 0.3 Ferrous materials are coated with aluminium by hot-dip aluminizing process primarily for protection against atmospheric corrosion and also for imparting to them resistance to high temperature oxidation and scaling. This recommended practice lays down the important steps which are required to be

followed for properly carrying out the process of hot-dip aluminizing of iron and steel articles.

- **0.4** In Appendix A, the information given in the last column aids inspectors in interpreting the appearance of the articles and helps them in arriving at a correct decision for rejecting the material not conforming to the relevant standard.
- 0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

- 1.1 This standard recommends important guidelines for general hot-dip aluminizing of iron and steel.
- 1.2 For guidance, a summary of defects commonly met in hot-dip aluminizing, marking conditions and safety measures which should be observed in the aluminizing plant have been given in Appendices A and B, respectively.

2. TERMINOLOGY

- 2.0 For the purpose of this standard, the following definitions shall apply.
- 2.1 Ash A mixture of aluminium oxide and varying quantities of metallic aluminium. The former is found as a result of oxidation of molten aluminium surface of the bath and when the oxide is skimmed off, a certain amount of metallic aluminium gets entrapped and is removed along with it.
- 2.2 Dross A complex mixture of aluminium and iron which forms in the aluminizing bath as a result of the reaction of molten aluminium with iron or iron salts and settles down at the bottom of the bath. Aluminium content in dross will vary between 70

- to 80 percent depending on the quantity of metallic aluminium entrapped in dross during its removal from the pot.
- 2.3 Flux A chemical compound or a mixture applied in the form of an aqueous solution and dried on to the work in the dry process or spread as a molten blanket over the aluminium bath in the wet process. The primary purpose of the flux is to help in keeping the surface of both work and molten aluminium free from oxide at the time of reaction.
- 2.4 Overpickling The undue attack of underlying ferrous surface by the pickling solution after the removal of the scale.
- **2.5 Inhibitor** A substance added to pickling solution to prevent undue attack on clean metal without affecting the scale removing property of the pickling solution.
- **2.6 Wetting Agent** A substance added to pickling solution to facilitate wetting of the work surface.

3. BASE METAL FOR ALUMINIZING

3.1 Steel — Mild steel (carbon steel with carbon 0.15 to 0.25 percent) is the most common material

that is aluminized, and the variations in the range of compositions used have little influence on the aluminizing process. The steel, however, should contain minimum amount of segregation, slag inclusions, rolled-in-mill scale, etc.

- 3.1.1 Carbon, silicon and manganese tend to decrease the rate of reaction between steel and molten aluminium. The effect, however, is not so apparent in the range of compositions encountered in mild steels as in high carbon and high silicon steels.
- 3.2 Cast Iron Cast irons react with aluminium differently depending on the exact composition, in particular, the silicon and phosphorus contents.

4. PREPARATION OF METAL SURFACE FOR ALUMINIZING

- 4.1 Cleaning If an article is contaminated by oilgrease or paint, pre-treatment in special solvents will be necessary for their removal. Several proprietary reagents are available. Alternatively, sodium hydroxide solution obtained by dissolving 10 to 15 kg of sodium hydroxide in 100 litres of water may be used.
- **4.1.1** The work should be handled in the degreasing bath so as to allow free circulation of liquid over all parts, taking care to clean the scum that may collect. The temperature of the solution should be usually kept between 85 to 90°C and the immersion time varying from 1 to 20 minutes depending on the nature and degree of contamination.
- **4.1.1.1** Immediately after degreasing, the work should be rinsed in hot water (60°C) followed, if possible, by a final rinse in cold running water.
- 4.1.2 When lubricating materials have reacted with the surface of the metal, it may be necessary to heat the part to bluing or scaling temperature in order to burn off the offending material. Since this is an expensive and cumbersome process, prior care should be taken to avoid such contamination.
- **4.1.3** Thermal cleaning may be adopted if the lubricating oils or grease evaporate easily without leaving any residue that may hinder pickling.
- 4.2 Cleaning of Castings Grey iron and malleable iron castings, if not properly cleaned before annealing, develop burnt-on sand patches at the surface which are not removed by normal pickling. Except in the case of light castings which would be damaged, all castings should be shot or grit blasted prior to aluminizing. It is generally recommended to give a quick rinse in dilute hydrochloric acid to wash off the grit dust. It is then processed according to normal fluxing practice.
- 4.2.1 An alternative but less efficient method of cleaning castings with burnt-on sand is to employ a pickle solution containing hydrofluoric acid. For use,

the commercial acid is diluted to various strengths, ranging from 1 volume of acid and 59 volumes of water for a very weak pickle to 1 volume of acid and 9 volumes of water for a very strong solution. With a weak solution, pickling may take as long as 24 hours while the strong solution should pickle satisfactorily in 10 to 30 minutes.

- 4.2.1.1 A hydrofluoric-acid pickle leaves a gelatinous layer on the surface of the castings which shall be thoroughly removed by hosing with water and scrapping and brushing. The casting shall then go through the oridinary pickling process.
- 4.2.1.2 Solution containing 6 parts by volume of commercial hydrofluoric-acid, 4 parts by volume of technical grade hydrochloric acid (see IS: 265-1987*) and 40 parts by volume of water may also be used for pickling. When pickling of castings is done occasionally, a solution consisting of 4 parts of dilute hydrochloric acid (1:1) and 1 part of hydrofluoric acid may be used.
- 4.3 Pickling Both hydrochloric acid (see 4.3.1) and sulphuric acid (see 4.3.2) solutions may be used for pickling. Hydrochloric acid is used at room temperature while with sulphuric acid, best results are obtained when it is hot (60 to 80°C).
- 4.3.1 Hydrochloric Acid Solution (150 g/1) Dilute technical grade acid conforming to IS: 265-1962* with an equal volume of water.
- **4.3.1.1** A suitable inhibitor should be used with hydrochloric acid. Iodine (0.2 percent m/v) may be used as inhibitor.
- 4.3.2 Sulphuric Acid Solution (100 to 150 g/1) Dilute 6 to 8 ml of technical grade acid conforming to IS: 266-1977† to 100 ml. The actual concentration of sulphuric acid solution, the temperature of the bath and the time of immersion will depend on the nature of work to be pickled.
- **4.3.2.1** A suitable inhibitor should be used with sulphuric acid.
- 4.3.3 Agitation Mild agitation of the work in the pickling tank reduces the time of pickling. Raise or lower the work once or twice to change the acid layer in contact with the work. Air agitation is not recommended.
- 4.3.4 Control of the Acid Solution To make the best use of the solution, reasonably close control of its acid content is necessary. The solution should be tested for acid and iron contents at regular intervals in accordance with the methods given in Appendix C. The strength of the solution should be maintained by periodic addition of fresh concentrated acid. The

^{*}Specification for hydrochloric acid (third revision).
†Specification for sulphuric acid (second revision).

iron salts in the pickling bath gradually accumulate with continued working and when the iron content reaches to about 100 to 120 g/l, the solution should be discarded.

- **4.3.5** Disposal of Waste Liquor The acid and iron compounds may be recovered from the waste pickling solution. Where this is not done, the pickling solution should be neutralized before dumping into sewers or streams. For this purpose, reference may be made to IS: 2490 (Part 1)-1981*.
- **4.4 Rinsing** After pickling, the article should be rinsed in running water. Two rinse tanks are preferable, the water cascading from one into the other.
- **4.4.1** If required, the articles after pickling may first be rinsed in line water before rinsing in running water as specified in **4.4**.

4.5 Prefluxing

- **4.5.1** The rinsed article in the dry process is dipped in a solution of suitable flux, such as potassium fluoride solution (10 percent, m/v). The fluxing solution may contain some wetting agent and the temperature may range from room temperature to 80° C.
- 4.5.1.1 When dry aluminizing is adopted, the article should be thoroughly dried after prefluxing in an air oven maintained at a temperature of about 120°C but not exceeding 130°C, as the flux is likely to crack and the base metal is liable to be oxidized.
- 4.5.1.2 In the molten flux process, a deep flux cover is used on the aluminium bath and the work in dry condition is immersed through the flux layer.
- 4.5.2 The article that has been pre-fluxed and dried should be aluminized without delay as the flux coating is likely to pull off and thus expose the work surface to atmospheric oxidation. The recommended practice is to aluminize immediately after fluxing and drying.
- 4.5.3 The concentration and volume of prefluxing solution should be maintained by addition of required quantities of flux crystals and water to make up for the drag-out losses.
- 4.5.4 It is also possible to aluminize ferrous materials by using organic fluxes (for example, linseed oil).

5. ALUMINIZING

5.1 Aluminium Used for Aluminizing — Aluminium conforming to Grade IV (99.0 percent) of

- IS: 2590-1987* should be used for general aluminizing work and aluminium 6 percent silicon alloy conforming to Grade 4300 (A-18) of IS: 617-1975† should be used for the purpose of imparting high temperature oxidation and scaling resistance.
- 5.2 Bath Temperature The control of bath temperature is essential if the quality of product is to be consistent and aluminium is to be used economically. Articles should be aluminized at the lowest possible temperature which will allow free drainage of aluminium from the work piece during withdrawal. A low temperature reduces the formation of ash and dross besides safeguarding the pot and conserving fuel. The bath temperature in case of commercially pure aluminium may vary from 680 to 720°C but in the case of aluminium bath containing suitable quantity of silicon and to some extent titanium, may be within 650 to 680°C.
- 5.3 Suspended Dross Dross should be allowed to settle at the bottom of the aluminium bath and should not be disturbed more than necessary during dipping operation. As far as practicable, the article should not be sunk to the bottom of the bath during aluminizing. Dross should be removed at least once every week preferably on the day following the weekly off-day or as often as necessary, when the temperature of the bath is lowered to slightly above the melting point, by means of a perforated scoop.
- 5.4 Rate of Immersion The work should be immersed as rapidly as possible but with due regard to operator's safety. This is specially important in wet aluminizing where the amount of dross increases with the time of contact with the flux blanket. The speed of immersion also influences the uniformity of coating, particularly with long work piece where the difference in immersion time between the first and the last part to enter the bath may be considerable.
- 5.5 Withdrawal The article shall be withdrawn slowly so as to allow the excess aluminium to flow down freely from the surface. It is better to use special jigs and carrier for dipping and withdrawing the works in batches.
- 5.5.1 Articles are withdrawn through a bath of clear aluminium to avoid contamination by flux.
- 5.6 Cooling Articles after being withdrawn from aluminium bath shall be air-quenched or quenched with sprayed water.
- 5.7 Centrifuging Small articles handled in baskets should be centrifuged to remove excess of aluminium immediately after aluminizing while the coating is

^{*}Tolerance limits for industrial effluents: Part 1 General limits (second revision).

^{*}Specification for primary aluminium ingots for remelting for general engineering purposes (second revision).

[†]Specification for aluminium and aluminium alloy ingots and castings for general engineering purposes (second revision).

still in the molten condition. The quality of finish depends on rapidity with which the material is transferred from the aluminizing batch to the centrifuge. It is also important that the centrifuge should be powered by a high starting torque electric motor to give rapid acceleration to peak speed within 2 to 3 seconds. After centrifuging, the article should be immediately dipped into water to allow the coating to set and prevent the articles from sticking to each other.

- 5.7.1 Thread Brushing Threads on articles which are unsuited for centrifuging shall be cleaned with a rotating wire brush immediately after aluminizing and before the coating sets. This process reduces the thickness and the protective value of the coating. It should only be confined to threaded portions of the article.
- **5.8** Stacking Articles should not be stacked immediately after cooling (see **5.6**) to avoid flaking of coating.

6. TESTING AND INSPECTION

- 6.1 Freedom from Defects The aluminium coating should be adherent, reasonably smooth, continuous and free from such imperfections as flux, ash and dross inclusions, patches and blisters.
- **6.2 Uniformity in Thickness** Aluminized articles should be tested for uniformity in thickness of

coating by any suitable method to be agreed upon between the supplier and the purchaser until an Indian Standard on the subject is published.

- **6.3 Mass of Coating** Mass of aluminium coating may be determined in accordance with IS: 3821-1973*.
- 6.4 Thickness of Coating Thickness of coating may be determined in accordance with the method prescribed in IS: 6697-1988†.
- 6.5 Adhesion Test The material should be tested in accordance with the relevant specification on the subject. Generally, however, for testing adhesion of aluminium coating, of sheet or wire, the sample in cold condition is bent through 180° over a mandrel of diameter equal to four times the sample thickness in the case of sheet and wrapped in a close helix around a mandrel of diameter equal to four times the sample diameter in the case of wire. After doing so, there should not be any cracking, peeling or flaking of the aluminium coating to such an extent that it may be removed by rubbing with bare fingers.

APPENDIX A

(Clauses 0.4 and 1.1)

DEFECTS, THEIR CAUSES AND REMEDIAL MEASURES

DEFECTS	CAUSES	RECOMMENDED ACTION	Guidance for Acceptance/ Rejection	
	Paint, grease, or oil residues	Check cleaning practices)	
Bare spots	Scale or rust residues	Check pickling practices		
	Residual welding slag	Blast-clean welds avoid coated rods	May be rejected except where bare spots are small and suitable for	
	Breakdown of preflux coating	Check preflux and drying conditions		
	Rolling defects in basis steel	Check steel supply	patching	
	Articles in contact during aluminizing	Keep articles separated	j	
General roughness	Analysis of original surface condition of steel	Check steel supply)	
	Overpickling	Reduce pickling, use inhibitor	May be accepted except by prior	
	High aluminizing tempera- ture or long immersion time or both	Adjust aluminizing conditions	agreement	

^{*}Methods for determination of mass of aluminium coating on hot-dip aluminized iron and steel articles.

[†]Specification for hot-dip aluminium coatings on ferrous parts (other than sheet, strip and wire) for general purposes (first revision).

DEFECTS		Causes	RECOMMENDED ACTION	Guidance for Acceptance/ Rejection	
Pimples		Entrapped dross particles	Avoid agitation or dross layer, check carry-over of pickle salts	May be accepted, unless dross conta- mination is heavy	
	ſ	Withdrawal speed too high	Remove work slowly)	
		Cold aluminizing bath	Increase temperature		
Lumpiness and runs (uneven drainage)		Delayed run-off from seams, joints, bolt holes, etc	Remove work slowly	Only by prior agreement	
		Articles in contact during withdrawal	Keep articles separated		
Flux inclusions	1	State flux burnt-on during dipping	Refresh or renew flux blan- ket if flux used on bath surface	May be rejected	
	ĺ	Surface residues on steel	Check steel preparation	}	
Ash inclusions	[Ash burnt-on during dipping	Skim bath before dipping	May be rejected, if	
Asii inclusions	{	Ash picked-up from top of bath	Skim bath before with- drawal	in gross lumps	
Black spots	{	Includes flux particles from flux 'dusting'	Confine fluxing to top of bath	May be rejected	
•	Ĺ	Dirt smuts, splash marks	Check storage conditions	May be accepted	
Dull grey coating		Slow cooling after aluminizing	Avoid hot stacking, quench	May be accepted if limited to occa-	
(all alloy, no free aluminium)	• {	Release of absorbed hydro- gen during solidification of coating	Avoid overpickling, use inhibitor	sional areas. Control by prior agreement	
Rust stains	{	'Oozing' of acid, from seams and folds	Check product design and fabrication	May be accepted	
		Storage near rusty material	Check storage condition	j	
		Expansion of entrapped hydrogen and moisture in flaws	Check steel quality		
Blisters	}	Driving off of hydrogen absorbed during pickling	Use short blast instead of pickle; Check steel supply	May be rejected, if general	
		Improper malleabilizing (for malleable iron castings only)	Check malleabilizing practice		
Tiny blisters		Effect sometimes absorbed on quenched work, notably malleable castings. May be caused by gas evolved from the work resulting from absorbed hydrogen or break-down of combined carbon near surface	Use shot blast instead of pickle Check malleabilizing treatment Should have no combined carbon near surface or casting	May be rejected, if blistering is generally widespread	

APPENDIX B

(Clause 1.1)

WORKING CONDITIONS AND SAFETY MEASURES

B-1. WORKING CONDITIONS

B-1.1 The aluminizing shop should be kept neat and tidy. Where possible, increased use should be made of hoods, extraction ducts and exhaust fans to give as good an atmosphere as possible.

B-2. SAFETY MEASURES

B-2.1 All safety measures should be properly exhibited.

B-2.2 The workers at the aluminizing bath should be provided with:

- a) eye or face shield,
- b) rubber boots,
- c) steel-capped boots,
- d) leather or leather on woollen base gloves,

- e) rubber and leather aprons, and
- f) long rubber or PVC or neoprene gloves.

B-2.3 While cleaning the articles with sodium hydroxide solution, the operators should be warned that it produces severe flash burns. Special precautions should be taken to protect them from splashes of sodium hydroxide solution.

B-2.4 Hydrofluoric acid sometimes used for pickling castings (see 4.2.1) is dangerous and causes very severe burns and sores when it comes in contact with the skin. It should, therefore, be carefully handled by wearing rubber boots, gloves and aprons.

B-2.5 Arrangement should be made to protect the workers from fumes over the aluminium bath.

B-2.6 So as to avoid any explosion in aluminizing bath, articles to be aluminized and tools used should be made completely dry, that is, free from moisture before dipping them in molten metal bath.

APPENDIX C

(Clause 4.3.4)

TEST METHODS

C-1. QUALITY OF REAGENTS

C-1.1 Unless otherwise specified, pure chemicals shall be employed in tests and distilled water (see IS: 1070-1977*) shall be used when the use of water as a reagent is intended.

Note — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

C-2. DETERMINATION OF ACID CONTENT OF THE PICKLING SOLUTION

C-2.1 Reagents

- C-2.1.1 Standard Sodium Carbonate Solution approximately 0.5 N.
- C-2.1.2 Methyl Orange Indicator Solution 0.05 g of methyl orange dissolved in 100 ml of alcohol.
- C-2.2 Procedure Filter exactly 25 ml sample of the pickle liquor into 250-ml measuring cylinder and make up to 250 ml by adding distilled water. Mix thoroughly. Pickle out 25 ml of this solution in

*Specification for water for general laboratory use (second revision).

a 250 ml concial flask. Add a few drops of methyl orange indicator and titrate it with the standard sodium carbonate solution to yellow end point.

C-2.3 Calculation

Mass of hydrochloric acid in g/1 = $\frac{A \times B \times 36.5}{2.5}$

Mass of sulphuric acid in g/1 = $\frac{A \times B \times 49}{2.5}$

where

A = volume in ml of the standard sodium carbonate solution required, and

B =normality of the standard sodium carbonate solution.

C-3. DETERMINATION OF IRON CONTENT OF THE PICKLING SOLUTION

C-3.1 Measure density of the pickling solution with a hydrometer, then on corresponding nomograph (see Fig. 1 and 2) depending on the acid used for pickling, join with a transparent ruler the point representing this reading shown on the left hand line with the point on the right hand line representing the acid content of the pickle.



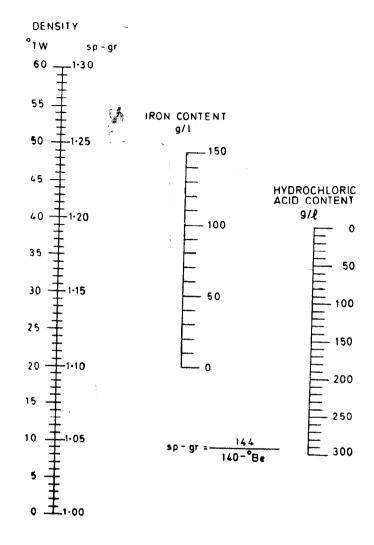


FIG. 1 Nomograph Relating the Iron Content of Hydrochloric Acid Pickle to the Acid Content and Density

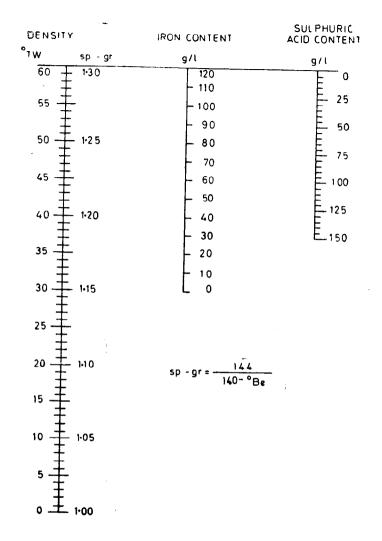


FIG. 2 NOMOGRAPH RELATING THE IRON CONTENT OF SULPHURIC ACID PICKLE TO THE ACID CONTENT AND DENSITY

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